

# **GaAs LPE GROWTH CENTRIFUGE - A NOVEL FACILITY TO PRODUCE HIGH PURITY GaAs MATERIAL**

Reinhard Katterloher, Gerd Jakob  
Max-Planck-Institut für extraterrestrische Physik, Postfach 1312, D-85741 Garching, Germany

Mitsuharu Konuma  
Max-Planck-Institut für Festkörperforschung, Heisenbergstr. 1, D-70569 Stuttgart, Germany

Alfred Krabbe  
University of California Berkeley, 366 Le Conte Hall #7300, Berkeley CA 94720, USA.

Nancy M. Haegel, S. A. Samperi  
Dept. of Physics, Fairfield University, North Benson Road, Fairfield, CT 06430-7524, USA.

Jeffrey W. Beeman  
Lawrence Berkeley National Laboratory, 1 Cyclotron Road, MS2-200, Berkeley CA 94720, USA

Eugene E. Haller  
UC Berkeley and Lawrence Berkeley National Laboratory, Berkeley CA 94720, USA

## **ABSTRACT**

GaAs photoconductive detectors will extend the spectral response cut-off up to  $\sim 310 \mu\text{m}$ . Progress in material research has led to the growth of pure, lightly and heavily doped n-type GaAs layers using the liquid phase epitaxy technique (LPE). Sample detectors have demonstrated the expected infrared characteristics of bulk type devices. Modeling of GaAs BIB detectors predicts a considerably improved IR sensitivity due to the higher doping of the infrared sensitive layer. However, the modeling gives also an estimate of the material requirements for the n-type blocking layer: Unintentional doping must stay below  $10^{13} \text{ cm}^{-3}$ . With a new centrifugal technique for the LPE material growth we intend to achieve high purity and reproducibility. Such a growth facility has been set-up at UCB one year ago. Contamination from outside during the LPE growth process is reduced by the suspension of the crucible on active magnetic bearings in a completely closed environment. A sequential combination of centrifugal and gravitational forces provides the proper transport of the Ga solution inside the growth crucible. Technical details of this unique equipment, first results of the achieved material quality in the initial growth runs and future steps to optimize operational parameters and material purity will be reported.

## **INTRODUCTION**

The long wavelength cut-off of bulk GaAs photoconductors around  $300 \mu\text{m}$  has been verified by photoconductive spectroscopy. Extending the cut-off even further and reducing the complexity of conventional FIR photoconductor arrays composed of single pixels (e.g., stressed Ge:Ga) can be achieved by developing planar integrated GaAs BIB arrays. Such arrays will require the successful growth of ultra-pure blocking layers and purely doped absorbing layers with exceptionally low minority dopant concentrations.

## **FIR GALLIUM ARSENIDE DETECTORS**

### **FIRGA detector array**

In the FIRGA project, which was sponsored by ESA, a 32 pixel GaAs array configuration has been developed<sup>1</sup>. The  $4 \times 8$  pixel array sensor was not composed of single pixels, but made from a monolithic

*Contact information for R. Katterloher: Email :rok@mpe.mpg.de, phone (49) (0) 89 30 000 3556*

multi-structured layer. The detectors were connected to two 18 channel cooled read-out electronics devices (CRE), fabricated by IMEC, Belgium. Because of the thin IR-active layer of 100  $\mu\text{m}$  the responsivity mean value was limited to 0.01 A/W. The low FIR responsivity was expected as the wavelength extension in bulk type photoconductors is always associated with a lower limit on the maximum allowed doping level before the onset of significant hopping conduction. The FIRGA demonstrator array, detector structure and response results are shown in Fig.1.

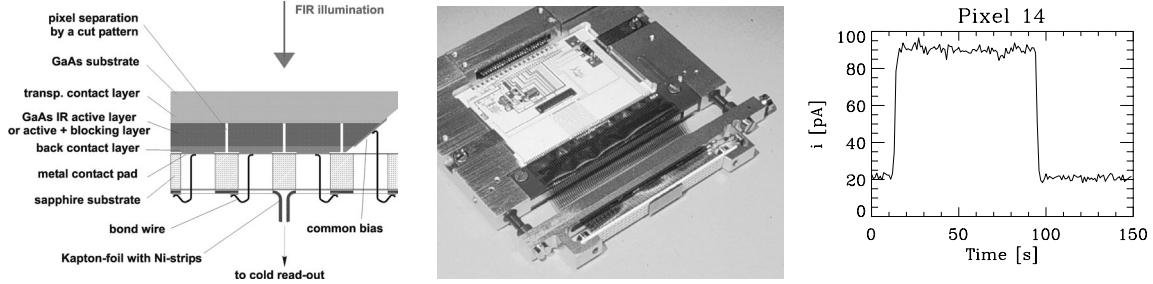


Figure 1: The FIRGA demonstrator array: Principle of the LPE grown detector structure and fan-out (left). FIRGA module with the 32 pixel (4x8) GaAs demonstrator array in the middle of the lower front side and CRE in the center (middle). Response transients at 1.9 K and 10 mV bias (right).

### GaAs BIB detector modeling

Modeling of a blocked impurity band (BIB) detector type predicts a considerably increased IR sensitivity due to the attainable higher doping of the infrared sensitive layer <sup>2</sup>. However, the modeling gives also an estimate of the severe material requirements for the n-type blocking layer: Unintentional doping must stay close to  $1 \times 10^{13} \text{ cm}^{-3}$ . When space charge effects in the blocking layer are included in the numerical simulation, the resulting deviation from a constant field approximation is remarkable as shown in Fig.2. Our future detector development program is aiming for realization of a BIB detector array.

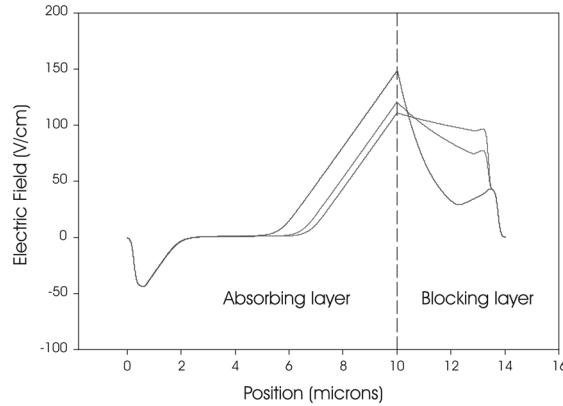


Figure 2: Effect of increasing doping in the blocking layer on the electric field distribution. Majority doping levels in the layer are  $5 \times 10^{13} \text{ cm}^{-3}$ ,  $1 \times 10^{14} \text{ cm}^{-3}$  and  $5 \times 10^{14} \text{ cm}^{-3}$  (lowest curve in the blocking layer). Detector bias voltage: 50 mV, temperature: 2.5 K.

## GaAs LPE MATERIAL GROWTH

### Liquid phase epitaxy

Liquid phase epitaxy (LPE), vapor phase epitaxy (VPE, including metal-organic compounds as sources) and molecular beam epitaxy (MBE) have been employed for growing GaAs layers. LPE appears to be the most promising technique for the growth of ultra-pure layers: The layers show high crystallographic perfection, high purity, and therefore, electrical properties close to the expected values <sup>4</sup>. Recently, investigations using cathode ray luminescence (CL) were performed on LPE and MBE produced samples. The LPE samples show significantly greater luminescence intensity at both room temperature and low

temperature. Spectra were taken with 20 or 25 keV electrons with penetration depths of about 2 to 4  $\mu\text{m}$ . Since the luminescence efficiency is proportional to the carrier lifetime, this is evidence that LPE grown layers are purer and will lead to better photoconductive devices. But, GaAs LPE material reproducibility in terms of control of both majority and minority doping (i.e., compensation) in sufficiently thick layers (using manually operated tipping boat techniques so far) needs further improvement to produce the high quality detectors required for state-of-the-art FIR astronomy.

### LPE centrifuge

With a new centrifugal technique for the LPE material growth we intend to achieve in a reproducible way the ultra high purity of a suitable blocking layer in a BIB device. Such a growth facility was put into operation at the Physics Department of UCB about one year ago. Contamination from outside during the LPE growth process is reduced by suspending the growth crucible on active magnetic bearings in a completely closed environment. A sequence of centrifugal and gravitational forces provides the proper transportation of the Ga solution inside the crucible. The graphite crucible has 4 inner chambers, 4 chambers in the middle for saturation and growth and 4 outer transfer chambers. Details of a sequence for a growth run on 1 substrate (step #5 in the schematic) are illustrated in Fig.3.

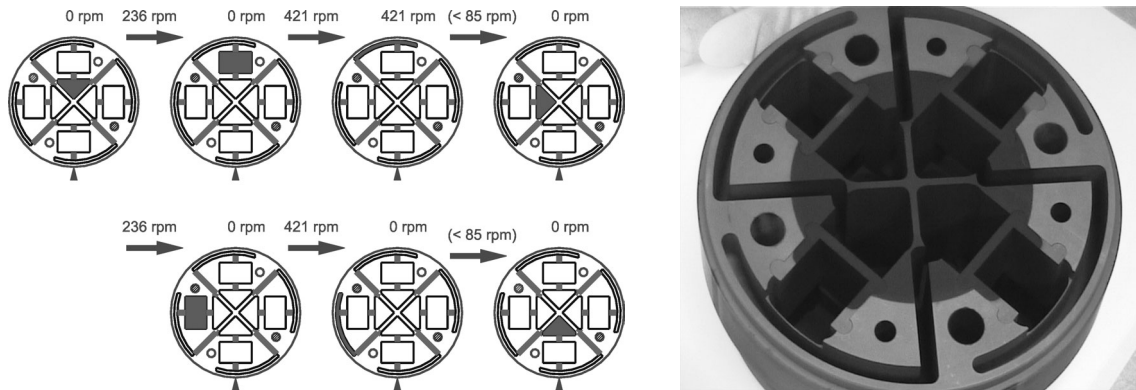


Figure 3: The growth crucible: Schematic of a transportation sequence of the Ga solution through the chambers in the crucible for a growth run on 1 substrate (left). Graphite crucible (right).

With the GaAs LPE centrifuge, multi-layers on substrates of increased size (presently 20mmx25mm) can be grown easily<sup>5</sup>. The system is highly automated and the growth run is completely computer controlled, thus assuring a high reproducibility of material quality for fixed operational parameters.

The LPE centrifuge assembly consists of the following basic elements:

- vacuum system (completely oil free vacuum pumps are used with a He-compressor supplied cryo-pump, vacuum tubing uses metal seals only)
- H<sub>2</sub> supply (4N H<sub>2</sub> is purified through a Pd diffusion cell and is flowing continuously through the growth reactor under slight overpressure)
- magnetic bearings carrying the rotor with the crucible (active control of 5 axes is performed, the rotor is driven by an inductive motor with speed control)
- growth reactor (different tubes made of SiO<sub>2</sub> or Al<sub>2</sub>O<sub>3</sub> can be used for the moment, the heating of the crucible is done by an external 3-zone furnace)
- cooling water loop

Main elements of the fully assembled centrifuge system and part of the control electronics are shown in Fig.4 on the right hand side.

An example of a growth program for production of two GaAs layers on two individual substrates is illustrated in Fig.4 (left side). The temperature timeline shows that growth takes place in two different temperature intervals, the corresponding rotation speed sequence required for proper transport of the Ga/GaAs solution inside the graphite crucible is inserted in the diagram.

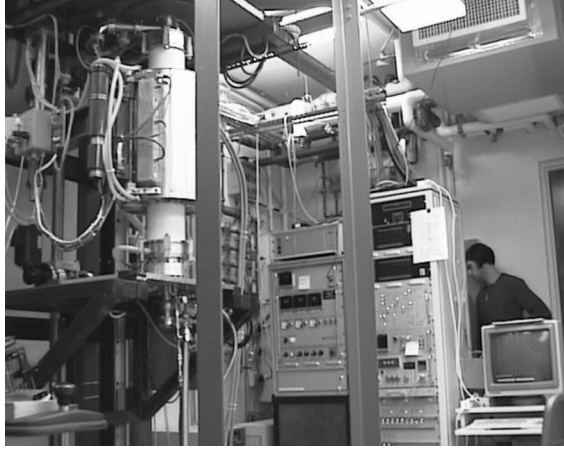
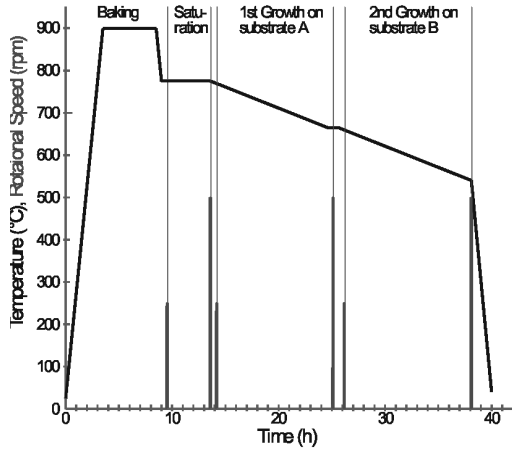


Figure 4: Temperature program and rotation sequence applied to grow GaAs layers on 2 substrates from 1 solution (left). LPE centrifuge assembly and control electronics at Physics Department of UCB (right).

### Growth run optimization

During the current experimental pre-growth development phase, we are working on reducing unwanted impurities<sup>3</sup>. We have achieved an improvement by an order of magnitude and we are making further progress. The first results are summarized in Fig.5. Hall measurements were performed on all layers grown to this day in order to determine net-carrier concentration and mobility versus temperature. The ongoing work will include technical improvements of the facility (e.g., air lock for crucible preparation), but the very next and main activities will concentrate on the further reduction of unintentional doping by more than one order of magnitude. Frequent and periodic bake-outs of the graphite crucible at high temperatures and cleaning of the growth reactor components with a number of chemical and thermal techniques are expected to lead to the required reductions of the majority and minority impurity concentrations.

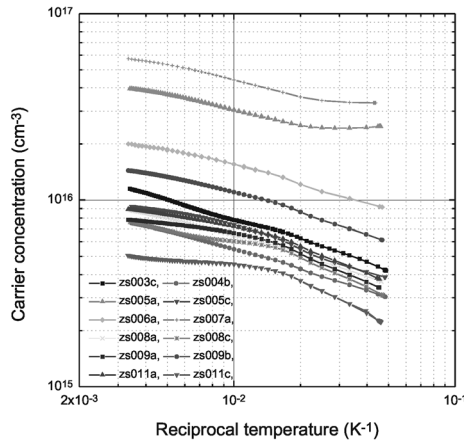


Figure 5: Unintentional net-carrier concentration in LPE GaAs layers produced during the initial growth runs (impurity species as identified by SIMS are mainly S and Si)

### REFERENCES

1. R. Katterloher, L. Barl, G. Jakob, M. Konuma, E. E. Haller, O. Frenzl, L. Hermans, "32 pixel FIRGA demonstrator – testing of a gallium arsenide photoconductor array for far infrared astronomy", *UV, Optical, and IR Space Telescopes and Instrumentation*, Proc. of SPIE Vol. **4013**, p. 100-108, Munich 2000
2. N. M. Haegel, J. E. Jacobs, and A. M. White, *Appl. Phys. Lett.* **77**, p. 4389, 2000
3. E. Czech, G. Götz, G. Cristiani, and M. Konuma, "Residual impurities in high purity GaAs layers grown by liquid phase epitaxy in H<sub>2</sub>-Ar atmosphere", *J. Cryst. Growth.* **198/199**, p. 1087, 1999.
4. S. Zehender, M. Konuma, I. Silier, E. Czech, E. Bauser, G. Jakob and R. Katterloher, Proc. 30th ESLAB Symp. "Submillimetre and Far-Infrared Instrumentation," ESTEC, ESA SP-388, p. 49, December 1996.
5. M. Konuma, I. Silier, E. Czech, and E. Bauser, "GaAs layers grown on 100 mm diameter Substrates in a liquid phase epitaxy centrifuge", In: *Gallium Arsenide and Related Compounds 1993*, Institute of Physics Conference, Series Number 136, ed. by H. S. Rupprecht and G. Weimann, Institute of Physics, Bristol and Philadelphia, p. 829, 1994.